

distillate to be collected in the graduated receiver in which the aqueous portion of the distillate is automatically separated and returned to the distillation flask.

4. Measure the volume of volatile oil which separates out as the upper layer in the graduated tube and calculate the %v/w on a dry weight basis.

Dried ripe fruits of caraway should contain not less than 2.5% v/w of volatile oil. The characteristics of the caraway oil are as given below:

|                    |                                     |
|--------------------|-------------------------------------|
| Colour             | : pale yellow                       |
| Odour and taste    | : aromatic and characteristic       |
| Weight (g) per ml  | : 0.90 to 0.91                      |
| Solubility         | : soluble in 8 parts of 80% alcohol |
| Optical rotation   | : at 25° + 70° to 80°               |
| Content of carvone | : 53 to 63% w/w.                    |

## 9.2 DETERMINATION OF ASH VALUES OF A CRUDE DRUG

Ash values are helpful in determining the quality and purity of a crude drug, especially in the powdered form.

The object of ashing vegetable drugs is to remove all traces of organic matter which may otherwise interfere in an analytical determination. On incineration, crude drugs normally leave an ash usually consisting of carbonates, phosphates and silicates of sodium, potassium calcium and magnesium. The total ash of a crude drug reflects the care taken in its preparation. Acid-insoluble ash is the residue left after extracting total ash or sulphated ash with HCl, while water-soluble ash represents that part of total-ash which is soluble in water. Water-soluble ash is good indicator of previous extract of water-soluble components of the crude drug. It also reflects on incorrect preparation or processing of crude drug. Acid-insoluble ash is indicative of siliceous or earthy material contamination of crude drug. A higher limit of acid-insoluble ash is imposed, especially in cases where silica may be present or when the calcium oxalate content of the drug is very high. Some analysts favour mixing of sulphuric acid with the powdered crude drug before ashing and this sulphated ash is normally less fusible than ordinary ash.

### Object

To determine the total ash and acid-insoluble ash of liquorice root.  
(Ref. I.P. 1985.)

### Materials

Silica crucibles, desiccator, ashless filter papers, dilute hydrochloric acid, powdered liquorice.

### Procedure

#### (a) Determination of total ash

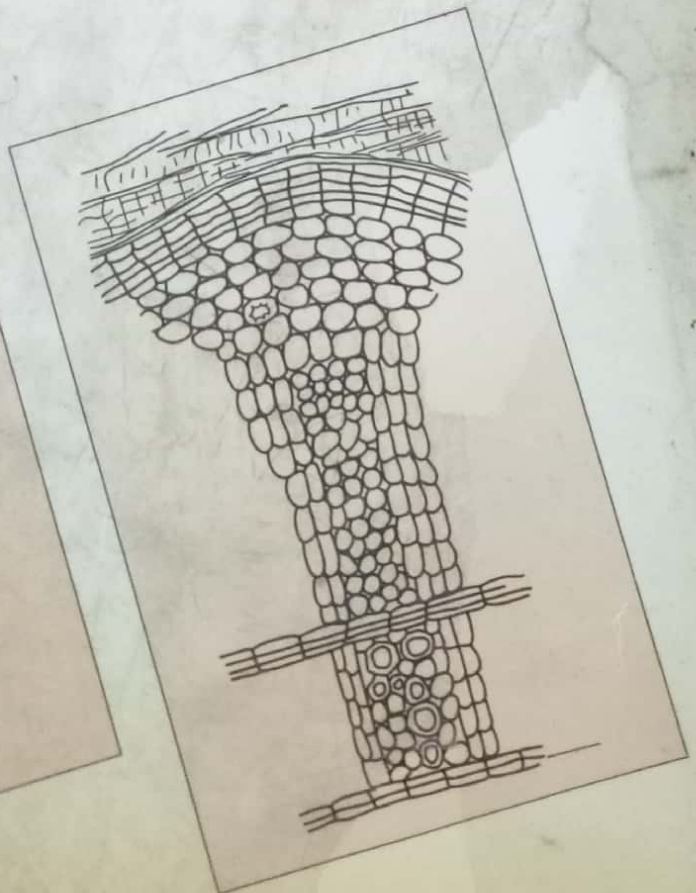
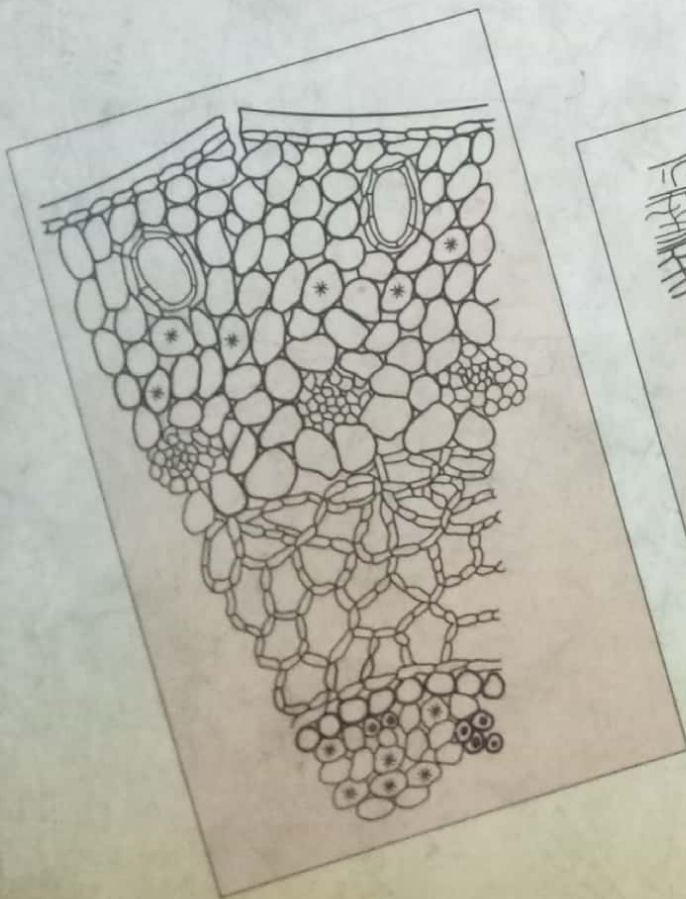
1. Weigh accurately about 3 g of the powdered drug in a tared silica crucible.
2. Incinerate the powdered drug by gradually increasing the heat until free from carbon and cool. Keep it in desiccator.
3. Weigh the ash and calculate the percentage of total ash with reference to the air-dried sample.

#### (b) Determination of acid-insoluble ash

1. Boil the total ash obtained as above for 5 minutes with 25 ml of dilute hydrochloric acid.
2. Filter and collect the insoluble matter on ashless filter paper, wash the filter paper with hot water, ignite in tared crucible, cool and keep in desiccator.
3. Weigh the residue and calculate acid-insoluble ash of liquorice with reference to the air-dried drug.

The unpeeled variety of liquorice root should contain not more than 10% of total ash and 2.5% of acid-insoluble ash.

# Practical Pharmacognosy



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